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ACCESSION NR: AP4016076

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AUTHOR: Wiatrowski, Wieslaw (Engineer)

TITLE: Protection against the effects of nuclear weapons

SOURCE: Przegląd techniczny, no. 4, 1964, 6

TOPIC TAGS: nuclear weapon, nuclear explosion, atomic bomb, radiation damage, radiation protection, radiation shielding, atomic blast effect, gamma radiation, neutron radiation, atomic shock wave, radiation shielding materials, hydrogen bomb, thermonuclear bomb, fission bomb, fusion bomb, radioactive fallout

ABSTRACT: The author describes in very general terms the nature and effects of nuclear weapons, which he classifies into explosive weapons and devices spreading radioactivity. The article concentrates on the first category, broadly describing the shock wave, the emission of light, heat, and penetrating radiation, and their effects on people and materials. Thicknesses of the latter which cut the radiation intensity by 50% are cited in view of their possible use in shielding. Radioactive fallout is also broadly discussed and some shielding properties of materials (air, wood, water, earth, concrete, iron, and lead) are given.

Card 1/1

WICH, Frantisek, inz.

Health considerations in selecting the site of enterprises with harmful exhalations. Hut listy 18 no. 81583-585 Ag '63.

1. Hutni projekt, Praha.

RODEWALD, W.J.; WICHA, J.

Aza-steroid alkaloids. Synthesis of A-Nor-E-homo-5-azacholestane. Bul chim PAN 11 no.8:437-441 '63.

1. Department of Organic Chemistry, University, Warsaw.
Presented by O. Achmatowicz.

RODEWALD, W. J.; WICHA, J.

Synthesis of A-nor-5-azacholestane. Bul chim PAN
12 no. 2: 95-98 '64

1. Department of Organic Chemistry, University,
Warsaw. Presented by O. Achmatowicz.

DUDEK, J., inz.; WICHARY, A.

Excerpts from the information on previous cooperation of the Miners' trade Union and the Association of Mining Engineers and Technicians elaborated by the Main Administrations of the organizations. Wiadom. gorn. 14 no.9:295-297 S'63

1. Sekretarz Generalny Zarzadu Glownego Stowarzyszenia Inzynierow i Technikow Gornictwa (for Dudek). 2. Sekretarz Zarzadu Glownego Zwiazku Zawodowego Gornikow (for Wichary).

Wicher, K.

MILGROM, P.; WICHER, K.

Immunologic study of disintegration processes in tuberculosis.
Med. dosw. mikrob., Warsz. 4 no. 2:227-246 1952. (CLML 22:4)

1. Of the Institute of Microbiology of Wroclaw Medical Academy
and of the Complex of State Sanatoria in Oborniki Slaskie.

Wicher, K.

MILGROM, F.; ZOPOTH, J.; WICHER, K.

Serological studies of tuberculosis. Med. dosw. mikrob., Warsz.
4 no. 3:351-352 1952. (GLML 23:3)

1. Summary of work progress presented at 11th Congress of Polish
Microbiologists held in Krakow May 1951. 2. Wroclaw.

WICHER, K

MILGROM, Feliks; WICHER, Konrad

Iconographia syphilidis experimentalis. Arch.im-mu-ter.dow.
2:185-197 1954.

1. Instytut Immunologii i Terapii Doświadczalnej PAN we Wrocławiu, (Dyrektor: prof. dr L. Hirschfeld) Dział Immunologii Szczegółowej (Kierownik: doc.dr F. Milgrom)
(SYPHILIS, experimental,
photographs of recent syphilis in rabbits)

MILGROM, Feliks; WICHER, Konrad

Reaction between serum and protein-precipitating substances as
a model of serological reaction. Arch.immun.ter.dosw. 2:127-134
1954.

1. Instytut Immunologii i Terapii Doświadczalnej PAN we Wrocławiu.
(Dyrektor: prof. dr L. Hirszfeld) Dział Immunologii Szczegółowej
(Kierownik: doc.dr F. Milgrom.

(SERODIAGNOSIS,

reaction between serum & protein-precipitating substances
as model of serol. reaction)

MILGROM, Feliks; WICHER, Konrad

Mass investigation of tuberculosis using Biernacki's reaction.
Arch.immun.ter.dosw. 2:173-184 1954.

1. Instytut Immunologii i Terapii Doświadczalnej PAN we Wrocławiu. Dyrektor: prof. dr L. Hirsfeld. Dział Immunologii Szczegółowej (Kierownik: doc.dr F. Milgrom)

(BLOOD SEDIMENTATION, in various diseases
tuberc.)

(TUBERCULOSIS, blood in,
sedimentation rate)

POLAND / Microbiology. Microbes Pathogenic for Man and F-4
Animals. Spirochaeta.

Abs Jour: Ref Zhur-Biol., 1957, No 17, 76884.

Author : Milgrom, Feliks; Wicher, Konrad; Matej, Henryk;
Rogala, Danuta.

Inst : Not given. - ON LAST PAGE

Title : Study of the Nature of Wassermann Antibodies.

Orig Pub: Przegl. dermatol. i wenerol., 1956, 6, No 5, 391-
396.

Abstract: A suspension of live or heat killed pallid spiro-
chetes (Nichols strain) isolated from the testicles
of rabbits was introduced internally (5 times in
the course of 8-10 days) to healthy rabbits and those
ill with syphilis. An increase of the titer of the
Wasserman reaction (WR) was noted in all of the
rabbits in 2-3 weeks after the introduction of the

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PROF. DR. F. MILGROM

CHIEF MEDICAL OFFICER

1. MARIA A. MARKSA 19.

WICHER, K.

EXCERPTA MEDICA Sec 15 Vol. 11/9 Chest Sent 58

2025. MICROMETHOD OF BIERNACKI'S REACTION IN MASS EXAMINATIONS -
Mikrometoda odczynu Biernackiego w badaniach masowych - Wicher K.
and Szwab J. Zakł. Mikrobiol. Śląskiej A.M., Zabrze-Rokitnica - POL.
TYG. LEK. 1957, 12/28 (1082-1083) Tables 1

The investigation of the sedimentation rate with this micromethod was done in 692 women. The blood was taken from the finger tips, mixed with natrium citricum in the test tube and drawn in a 0.45-mm. pipette up to 20 cm. Increased ESR was found in 82 cases, 9 of which had active tb. The authors' suggestion to use this method for the purpose of detecting cases of tb does not seem right, as the character of the above reaction is not specific and it cannot replace mass X-ray investigation.

Węgrzynowska - Cracow

LESINSKI, Janusz; WICHER, Konrad; SPETT, Janina; ZAJAC, Wieslaw

Studies on the appearance of immobilizing antibodies in guinea
pigs. Postepy hig. med. dozw. 13 no.3:319-321 1959.
(ANTIBODIES) (SYPHILIS, immunol.)

WICHER, Konrad; LESINSKI, Janusz; JAKUBOWSKI, Adam

Studies on the course of experimental syphilis in guinea pigs.
Polski tygod. lek. 14 no.26:1218-1219 29 June 59.

1. (Z Zakładu Mikrobiologii Śląskiej A. M. w Zabrzu, p.o. kierownik:
kand n. med. K. Wicher i z Kliniki Dermatologicznej A. M. w Białymstoku:
kierownik: doc. dr J. Lesinski).
(SYPHILIS, exper.)

WICHER, Konrad; ROGALOWA, Danuta

Studies on acquired resistance of *Treponema pallidum*. Med.dow.
mikrob. 12 no.4:383-388 '60.

1. Z Zakładu Mikrobiologii Sl. A.M. w Zabrze-Rokitnicy p.o.
Kierownika Zakładu dr K.Wicher.
(SYPHILIS exper)

WICHER, Konrad; WOZNICZKO-ORLOWSKA, Genowefa

Attempted demonstration of differences of the group iso-antibody titer in children in relation to the maternal blood group. Polski tygodn.lek. 15 no.13:481-482 28 Mr. '60.

1. Z Zakładu Mikrobiologii Sl. A.M. w Zabrzu-Rokitnicy; p.o. kiero-
wnika: dr. Konrad Wicher.
(BLOOD GROUPS)

CZECHOSLOVAKIA

WICHTERLE, I.

Institute of Chemical Process Fundamentals of the Czechoslovak Academy of Sciences, Prague-Suchbát

Prague, Collection of Czechoslovak Chemical Communications,
No 10, 1965, pp 3388-3397

"Liquid-Vapour Equilibrium. XXXV. Vapour-Liquid Equilibria
in System Heptane-Toluene-p-Xylene and in Systems Heptane-
Toluene-Extractive Agent."

KICZAK, Janina; WICHERT, Krystyna

A cured case of agranulocytosis caused by gold therapy. Pol. arch.
med. wewn. 33 no.1:85-90 '63.

1. Z II Kliniki Chorob Wewnętrznych AM w Szczecinie Kierownik: prof.
dr med. E. Gorzkowski.
(AGRANULOCYTOSIS) (GOLD) (ARTHRITIS, RHEUMATOID)

KICZAK, Janina; WICHERT, Krystyna

Analysis of blood coagulation disorders in 16 cases of plasmocytic reticuloma. Roczn. Pom. akad. med. Swierczewski 10:419-432 '64.

1. Z II Kliniki Chorob Wewnętrznych Pomorskiej Akademii Medycznej (Kierownik: prof. dr med. Edward Gorzkowski).

KICZAK, Janina; WICHERT, Krystyna

Apropos of the palliative treatment of renal insufficiency with
a Lespedeza capitata extract (lespenephryl). Pol. tyg. lek.
19 no.32:1238-1240 10 Ag '64.

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Medycznej w Szczecinie (kerownik: prof. dr med. Edward
Gorzkowski).

KICZAK, Janina; WICHERT, Krystyna

Further studies on fibrinolysis in rheumatism. Pol. arch. med.
wewnet. 35 no.5:633-638 '65.

1. Z II Kliniki Chorob Wewnętrznych Pomorskiej Akademii Medycznej
w Szczecinie i z Ośrodka Klinicznego Sanatorium "Gryf" w Polczynie
Zdroju (Kierownik: prof. dr. med. E. Gorzkowski).

KICZAK, Janina; BRANDOWSKA, Maria; STALEWSKI, Ryszard; WICHERT, Krystyna

Studies on fibrinolysis in leukemias. Pol. arch. med. wewnet. 35
no.6:785-792 '65.

1. Z II Kliniki Chorob Wewnetrznych Pomorskiej AM w Szczecinie
(Kierownik: prof. dr. med. E. Gorzkowski) i z I Kliniki Chorob
Wewnetrznych Pomorskiej AM w Szczecinie (Kierownik: doc. dr. med.
K. Gregorczyk).

KICZAK, Janina; EISNER, Marek; BURA, Helena; WICHERT, Krystyna

Studies on blood coagulation and fibrinolysis in thyroid diseases.
Pol. arch. med. wewnet. 35 no.9:1337-1342 '65.

1. Z II Kliniki Chorob Wewnętrznych Pomorskiej AM w Szczecinie
(Kierownik: prof. dr. med. E. Gorzkowski), z III Kliniki Chorob
Wewnętrznych Pomorskiej AM w Szczecinie (Kierownik: doc. dr. med.
M. Eisner) i z I Kliniki Chorob Wewnętrznych Pomorskiej AM w
Szczecinie (Kierownik: doc. dr. med. K. Gregorczyk).

WICHLINSKI, Leslaw

Studies on retro-isomerization of ergot alkaloids. Acta Pol.
pharm. 22 no.3:237-241 '65.

1. 2. Laboratorium Badawczego Farmaceutycznej Spoldzielni Pracy
"Filofarm" w Bydgoszczy (Kierownik: dr. J. Trzebinski).

WICHLINSKI, Leslaw, dr.farm.

Contemporary problems of research on ergot. Farmacja Pol 18 no.4:
84-86 P '62.

1. Laboratorium Badawcze, Farmaceutyczna Spoldzielnia Pracy
Filofarm, Bydgoszcz.

POLAND

WICHLINSKI, L. [affiliation not given]

"Problems of the Stability of Drugs in the Light of Recent Investigations."

Warsaw, Paracelsus Polaka, Vol 18, No 23, 10 Dec 62, pp 561-564

Abstract: Instability of drug preparations is generally attributed to: hydrolysis, oxidation, racemisation, presence of metallic catalysts, influence of light, microbial agents; variation in pH and the nature of packaging materials. The methods of overcoming these difficulties are reviewed and a rapid method of determining stabilities of preparations is described.

This article contains eighteen references. Fourteen of the references are western.

1/1

WICHLINSKI, S.

Treatment of bleeding during menopause by steam. Przegl. lek.,
Krakow 8 no. 9:265-269 1952. (CML 23:5)

1. Of the Obstetric-Gynecological Department (Head--M. Glowinski,
M. D.) of Bytom Municipal Hospital No. 1.

WICHNEROVA, Eva; MISAK, Jan; Technicka spoluprace MOJZISOVA, Zdenka

Function examinations of the pancreas. I. Our experiences with function tests of the pancreas by the method of 2 sounds in chronic diseases of the pancreas and in some other chronic diseases of the neighboring organs. Cas. Lek. Cesk. 101 no.16/17:535-538 27 Ap '62.

1. II klinika nemoci vnitřních LFH KU v Praze, prednosta prof. dr. J. Syllaba, Dr.Sc., oddelení pro klinickou biochemii fakultní nemocnice v Praze 10, prednosta MUDr. RNDr. J. Oppl. t.

(PANCREASES diseases)	(GALLBLADDER diseases)
(PEPTIC ULCER diagn)	(PANCREATIC JUICE chem)

WICHRZYCKA, Elzbieta

Lymphocyte — a still unknown cell. Polski tygod. lek. 15 no.47:
1813-1816 21 N '60.

1. Z Oddziału Hematologicznego Instytutu Hematologii w Warszawie;
ordynator Oddziału: dr med. S. Pawelski; dyrektor Instytutu: doc.
dr med. A. Trojanowski.

(LYMPHOCYTES)

PAWELSKI, Sławomir; WICHRZYCKA, Elżbieta; MIZIEMSKI, Bohdan; ROSZEWSKI, Stanisław

Behavior of alkaline phosphatase in granulocytes of chemical workers and radiologists. Pol. tyg. lek. 19 no.38:1433-1435
21 8 '64

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WICHRZYCKA, Elzbieta

PAS reaction in proliferative diseases of the lymphatic system.
Pol. tyg. lek. 20 no.29:1076-1078 19 J1 '65.

1. Z Kliniki Chorob Wewnętrznych Instytutu Hematologii (Kierownik:
doc. dr. med. S. Pawelski).

WICHRZYCKA, Elzbieta

Attempted classification of leukemias according to the glycogen content of mother cells. Pol. arch. med. wewnet. 35 no. 8:1271-1275 '65.

1. Z Oddzialu Chorob Wewnetrznych Instytutu Hematologii w Warszawie (Kierownik: doc. dr. med. S. Pawelski).

ROSZKOWSKI, Ireneusz; KRETOWICZ, Janusz; WICHRZYCKI, Andrzej

Attempted clinical application of fetal electrocardiography and
phonocardiography. Pol. tyg. lek. 17 no.34:1325-1329 20 Ag '62.

I. Z II Kliniki Poloznictwa i Chorob Kobietych AM w Warszawie;
kierownik: prof. dr med. Ireneusz Roszkowski.

(FETAL HEART) (ELECTROCARDIOGRAPHY)
(PHONOCARDIOGRAPHY)

ROSZKOWSKI, Ireneusz; KRETOWICZ, Janusz; WICHRZYCKI, Andrzej

Evaluation of the usefulness of electrocardiography and
phonocardiography in establishing fetal life during the
2d and 3d trimester of pregnancy. Ginek. pol. 34 no.2:
189-192 '63.

1. Z II Kliniki Położnictwa i Chorob Kobietych AM w Warszawie
Kierownik: prof. dr med. I. Roszkowski.

(FETAL HEART) (FETAL DEATH) (DIAGNOSIS)
(ELECTROCARDIOGRAPHY) (PHONOCARDIOGRAPHY)

WICHRZYCKI, F.

The teaching program in the secondary school for road builders. p. 215.

DROGOWNICTWO, Vol. 10, No. 9 Sept. 1955

(Instytut Techniki Budowlanej) Warszawa

SOURCE: East European Accessions List Vol. 5, No. 1

Jan. 1956

WICHTERLE, F.
/ Liquid-liquid equilibrium. I. Dependence of the partition coefficient of one component on the phase compositions in a ternary system where two components are immiscible in the liquid phase. I. Wichterle and B. Follprechtová (Vysoká škola chem.-technol., Prague). *Collection Czechoslov. Chem. Commun.*, 25, 2492-6 (1960) (in German).—The partition coeff. of a component between 2 immiscible liquids is expressed by the 2-suffix Margules equation. From the solubilities of that component in the 2 solvents and from its ideal soly. it is possible to calc. the partition coeff. as a function of the compns. in the 2 conjugated phases. The distribution of I between the H_2O and the CCl_4 at 25° is given as an illustration. E. Erdős

WICHTERLE, I.; FOLLPRECHTOVA, B.

Equilibrium of liquidity-liquidity. I. A study of the dependence of the distribution coefficient of a component on the phase composition of a three-component system in which two components are immiscible in the liquid phase. Coll Cz chem 25 no.10:2492-2496 0 '60.

(EEAI 10:9)

1. Technische Hochschule fur Chemie, Prag.

(Systems(Chemistry)) (Phase rule and equilibrium)

WICHTERLE, Ivan

Extractive distillation. Chem listy 58 no.2:142-162 F '64.

1. Ustav fyzikalni chemie, Ceskoslovenska akademie ved, Praha.

KRISTEK, A.; KONIG, B.; WICHTERLE, O.

Contribution to the surgery of retinal detachment. Our experience with ethylenglycolmetacrylate gel plugs. Preliminary report. Cesk. oftal, 22 no.1:58-61 Ja '66.

1. Oční klinika lékařské fakulty Palackého University v Olomouci (prednosta: prof. dr. V. Vejdovsky, DrSc.) a Ústav makromolekulární chemie Československé akademie věd v Praze (reditel: akademik O. Wichterle).

The products of addition of hydrocyanic acid to glucosylarylamines (and other aldose arylamines) and glucosylpiperidines. R. Votček and O. Wichterle. *Collection Czechoslov. Chem. Commun.* 9, 109-10 (1937).—The addn. of HCN to a series of arylamino and piperidino derivs. of pentoses and hexoses was made for the purpose of (a) sapon, the resulting nitriles to the corresponding acids and reducing the lactones to arylamino sugars of the chitosamine type and (b) converting the ether-sol. derivs. of the nitriles by means of Grignard reagents to C-alkylated keto sugars. In the case of (a), the instability of the arylamino nitriles resulted in partial removal of the amino group and profound decompn. as evidenced by the presence of PhNC odor. Heating 20 g. d-xylose, 15 g. PhNH₂ (I) and 45 cc. abs. EtOH to a clear soln. followed by cooling gave 22.5 g. d-xylosylphenylamine (II) m. 148°, $[\alpha]_D^{25}$ initial value by extrapolation, -79.6°, final value -24°. A soln. of 2 g. l-fucose and 1.5 g. (I) in 10 g. abs. EtOH boiled for 5 min. gave upon cooling 2.6 g. l-fucosylphenylamine (III), m. 180-1°, $[\alpha]_D^{25}$ initial value by extrapolation, 102°, final value, 49°. A mixt. of 1.8 g. d-glucose and 1.4 g. m-NO₂C₆H₄NH₂ heated to 130-40° yielded upon cooling 1.0 g. d-glucosyl-m-nitrophenylamine, m. 172-80°. The following nitriles were prepd.: phenylamino-l-arabohexonic, obtained in 4 g. yield by heating 5 g. l-arabinose and 4 g. I in 6 g. abs. EtOH and, without isolating the condensation product, adding 10 g. abs. EtOH and 2 g. anhyd. HCN, crystals from abs. alc., m. 150° (decompn.) $[\alpha]_D^{25}$ -157° (1% MeOH soln.); phenylamino-d-xylohexonic, from 5 g. II and 6 cc. 37% HCN in 50 cc. abs. EtOH at ordinary temp., m. 115-20°; phenylamino-l-rhamnohexonic, obtained in 5 g. yield by heating 10 g. rhamnose and 7.5 g. I in 100 cc. abs. EtOH and, without isolating the amino deriv., adding 25 cc. 37.8% HCN and 15 cc. 94% EtOH, crystals from abs. alc., m. 143°, $[\alpha]_D^{25}$ -34.5° (MeOH soln.); phenylamino-l-fucohexonic, obtained in 1.0 g. yield by treating 2.2 g. III with 3 cc. anhyd. HCN, crystals from abs. alc., m. 173-4° (decompn.), $[\alpha]_D^{25}$ 184° (0.5% MeOH soln.); phenylamino-d-mannoheptonic, obtained by treating 2 g. mannosylphenylamine with 7 cc. 37.8% HCN in 200 cc. 94% EtOH, $[\alpha]_D^{25}$ 150° (0.5% MeOH soln.); piperidyl-l-rhamnoheptonic, obtained by boiling for 10 min. a mixt. of 5 g. l-rhamnose, 3 g. (CH₃)₂NH (IV) and 10 cc. abs. EtOH and adding in the cold 3 cc. anhyd. HCN, crystals from abs. alc., m. 142-3°, $[\alpha]_D^{25}$ 27° (1% MeOH soln.); piperidyl-d-mannoheptonic, obtained by heating 3 g. d-mannose, 3 g. IV and 10 cc. abs. EtOH followed by adding in the cold 5 cc. anhyd. HCN, crystals from abs. alc., m. 125-7°, $[\alpha]_D^{25}$ -10° (1% MeOH soln.). Heating a suspension of 3 g. phenylaminoglucoheptonic nitrile (V) and 3 g. AcONa in 12 cc. Ac₂O, followed by a treatment with cold H₂O and aq. Na₂CO₃ soln. and triturating with H₂O, yielded 5.3 g. of the corresponding penta-Ac deriv. of V. Similarly, treating 4 g. of phenylaminogalactoheptonic nitrile (VI) and 4 g. AcONa in 15 cc. Ac₂O yielded 7.5 g. of the corresponding penta-Ac deriv. of VI, m. 122°. I, P. L.

A 54-514 METALLURGICAL LITERATURE CLASSIFICATION

1ST AND 2ND DEGREE										3RD AND 4TH DEGREE									
PROCESS AND PROPERTIES INDEX																			
3C										A-3									
<p>Synthesis of di-xyloxyethylenic acid. O. WIGNER (Coll. Czech. Chem. Comm., 1933, 253-256).—Oxidation (KMnO₄ at 0°) of di-Ca α-hydroxy-Δ²-pentenoate and acetalization yields di-xyloxyethylenic acid (bromine salt, m.p. 183-184°, [α]_D²⁵ -25.6° in H₂O) which readily lactonizes on evaporation, and is oxidized (HNO₃) to di-tartaric acid; it could not be epimerized by C₂H₅N to the acid obtained by oxidizing 3-angulicactone (Thiele et al., A., 1902, 1, 156).</p> <p>A. Li.</p>																			
ASB-11A METALLURGICAL LITERATURE CLASSIFICATION										USONH NOMINAT									
USONH NOMINAT										USONH NOMINAT									

OPEN MATERIAL NOTE		PROCESSES AND PROPERTIES INDEX	
<div style="position: relative;"> CO <p style="margin-top: 100px;"> β-Chlorobutyric acetals. O. Wichterle and I. Vavrečka. <i>Collection Czechoslov. Chem. Commun.</i> 10, 403-6(1938). To 100 cc. MeOH satd. with dry HCl was added 100 g. calcined Na₂SO₄, then with violent agitation and cooling was added dropwise 500 cc. MeCH:CHCHO (I), the temp. being maintained below 5°. The lower layer was agitated with K₂CO₃ soln. (II), then treated with NaHSO₄ (III) and again repeatedly with II, and dried over solid II. β- <i>Chlorobutyric di-Me acetal</i> (IV) (580 g.), b_m 55-7°, was ob- tained. I (178 g.) was added to a well-cooled and agitated soln. of PrOH satd. with HCl, the temp. being kept below 5°. The lower layer was poured into ice water and washed immediately with II, then with III and dried over solid II. The β-chlorobutyric di-Pr acetal thus obtained b_m 102-4°. β-Chlorobutyric di-Bu acetal, b_m 130-1°, and the diiso-Bu acetal, b_m 120-30.5°, were prepd. in the same way. <div style="text-align: right;">I. C. Lo Cicero</div> </p> </div>		<div style="text-align: right; font-size: 1.5em; margin-bottom: 10px;">10</div>	
ASD-SLA METALLURGICAL LITERATURE CLASSIFICATION		OTHER DIVISION	
SYMBOLS FOR ELEMENTS		SYMBOLS FOR COMPOUNDS	

1-Alkoxybutadienes. O. Wichterle. Collection Czechoslov. Chem., Commun. 10, 497 (1938). — $\text{MeCHClCH}_2\text{CH}_2\text{OEt}$ (I) (210 g.) purified with NaHSO_4 and by distn. In vacuo was heated with PbMe_2 . The vapors were led through a Hempel column with PhMe. The Hahn column was led through a distn. stopped. The Hahn column (100°) was collected; the distn. moved with II (1) to a const. vol. was removed and 13 cc. more was washed with II (1) to a const. vol. combined distillates were washed with K_2CO_3 and fractionated, giving 3 fractions, 4.6 g. b. 80–111.5°, 40 g. b. 111.5–12°, 14 g. b. 112–48°. Residn. in vacuo eliminated decompn. as evidenced by residues in the flask and column. The 2nd fraction was 1-alkoxy-1,3-butadiene (II), the 3rd on re-distn. gave a fraction b. 145–8°, identified as crotonic aldehyde. The proportions of the 2 formed under practically identical conditions varies considerably, thus indicating that the course of the reaction is probably influenced by accidental catalysts. II (9.8 g.) and 6.5 g. $\text{CH}_2\text{CHClCH}_2\text{OEt}$ (III), both freshly distd., were heated 3 hrs. in a sealed tube on a water bath. Distn. in vacuo, after excess III was eliminated, gave 1 g. b. below 91° , 2.5 g. b. 91– 100° , and a residue. Repeated fractionation gave 2-fractions: Δ^2 -tetrahydrobenzaldehyde (IV) b. 90°. II (8 g.) and 11 g. $\text{MeCHClCH}_2\text{OEt}$ (V), both freshly distd., were heated 6 hrs. in a sealed tube 7 g. 6-methyl-2-(5)-ethoxy- Δ^2 -tetrahydrobenzaldehyde (VI) (100 g.) was refluxed with 400 g. fractionation in vacuo gave 7 g. b. 120–70°, washed with K_2CO_3 and fractionated. The soln. was distd. (120–70°), washed with Na alloyate (VIII) (for removal of K_2CO_3). Fractionating gave 8 g. 1-propoxy-1,3-butadiene (IX), reducing the vol. to 35 cc., and dried over CaH_2 . Fractionation gave 8 g. 1-propoxy- Δ^2 -tetrahydrobenzaldehyde (X), along with 8.5 g. crotonic aldehyde, b. 116–18°, 35.5–6.5°, along with 1,1,3-tripropoxybutane, b. 76–77° and 7.5 g. III, both freshly distd., were heated 3 hrs. in a sealed tube at 100° . Double fractionation gave 15 g. 2-(5)-propoxy- Δ^2 -tetrahydrobenzaldehyde (XI), b. 103–4°, VIII (10 g.) and 12 g. V, both freshly distd., were heated 3 hrs. in a sealed tube at 145–55°. Several fractionations in vacuo gave 3 g. 6-methyl-2-(5)-propoxy- Δ^2 -tetrahydrobenzaldehyde (XII), and 112–15°. 8-Chlorobutyric di-Bu acetal (XIII) (110 g.) and 400 g. KH were refluxed and the soln. was then distd. The 100 cc. collected was agitated with VIII, reducing the vol. to 60 cc., then dried over K_2CO_3 and fractionated.

SEE OTHER SIDE

twice in vacuo. A 33% yield of 1-butoxy-1,3-butadiene (XIV), b_p 53.5-4.5°, was obtained. Crotonic di-*iso*-Bu acetal, b_p 103-4°, was obtained, along with XIV. XIV (16 g.) and 18 g. V, both freshly distd., were heated 4 hrs. in a sealed tube at 140-50°. Several fractionations gave 15.4 g. 6-methyl-2(5 β)-butoxy- Δ^4 -tetrahydrobenzaldehyde, b_p 127-0°. *n*-Chlorobutyric di-*iso*-Bu acetal (120 g.) was refluxed with 400 g. KOH and treated as before. Isobutoxybutadiene (XV) (27.5 g.), b_p 53-6°, was obtained along with 5.8 g. crotonic di-*iso*-Bu acetal, b_p 103.5-4.5°. XV (24.5 g.) and VIII were heated 5 hrs. in a sealed tube at 140-50°. Several fractionations in vacuo gave 20 g. 6-methyl-2(5 β)-isobutoxy- Δ^4 -tetrahydrobenzaldehyde. J. C. Lo Cicero

1ST AND 2ND ORDERS

3RD AND 4TH ORDERS

10

PROCESSES AND PROPERTIES INDEX

A

A methylenetetrahydroxyadipic acid. H. Votoček and O. Wichterle. *Collection Czech. Chem. Commun.* 11, 266-71 (1930).—Oxidation of α-rhamnobrexonic acid with HNO₃ yields 6-keto-α-rhamnobrexonic acid (I) (C. A. 26, 4306). The Ba salt, prepd. from 17.6 g. I, was treated in 170 cc. H₂O with 7 cc. of anhyd. HCN. After 13 hrs. the faintly brown gelatinous product was taken up in 21 H₂O, boiled and sepd. by boiling for several hrs. with excess Ba and soapon. The excess Ba(OH)₂ was removed with CO₂ and (OH)₂. The excess Ba(OH)₂ was removed with CO₂ and the filtered liquid was concd., acidified with H₂SO₄ to tropocollin. Crystn. of the 21 g. of sirup from alc. gave 9 g. of pentahydroxypimelic lactone (II), C₁₁H₁₆O₈, m. 195°, [α]_D²⁰ -20.4°, forming a cryst. Ba salt, C₁₁H₁₀BAO₈·4H₂O. II does not reduce Fehling soln. The non-crystallizable portion of the sirup is mainly the epimeric acid. To decide between the 2 possible structures, attempts were made to transform II into the corresponding methylpenta- to me. II (3.7 g.) in a small amt. of H₂O at 0° was acidified with H₂SO₄ and shaken with 60 g. of 3.6% Na-Hg lastro- with H₂SO₄ and shaken in 5 hrs. The liquid was made faintly acid and shaken for 15 days until the reducing power to Fehling soln. disappeared. The soln. was acidified with H₂SO₄ to tropocollin, concd. until the Na₂SO₄ crystd. out, and pptd. with excess abs. alc. The sirup was dissolved in 100 cc. of ice, acidulated H₂O and reduced at 0° with Na-Hg under faintly acid conditions. After 8 hrs. the Na₂SO₄ was removed and the sirup was treated with excess PhNHNH₂·AcOH, yielding a yellow gelatinous osazone. Dry distn. of II produced an oil which gave a green color with FeCl₃, characteristic of pyrone derivs. The lactone ring is thus due to loss of H₂O between a CO₂H group and a 4-OH group.

C. R. Addinall

METALLURGICAL LITERATURE CLASSIFICATION

FROM SYMBOLIC

SUBORDINATE

FROM SYMBOLS

SUBORDINATE

CA

10

The preparation of 1-ethoxy-1,3-butadiene by the addition of ethanol to vinylacetylene. O. Wichterle and J. Procházka. *Chem. Listy* 36, 278-80(1942).—The product of the addn. of EtOH to $\text{CH}_2\text{=CHC}\equiv\text{CH}$ (I) was identified as 1-ethoxy-1,3-butadiene by means of mol. refraction. A 25% soln. (160 g.) of I in EtOH was heated 10 hrs. in an autoclave with 110 g. KOH to 140-180° (pressure, 50 atm.), the mixt. steam-distd., the distillate acid. with K_2CO_3 , and the sepd. layer dried with K_2CO_3 ; after stripping off the EtOH, distn. through a 40-cm. Widmer column gave about 10 ml. of a fraction b. 109-12° which, after repeating the fractionation, had d_4^{20} 0.8830, n_D^{20} 1.44813, 1.45287, 1.45554, 1.47822 for C, D, E, and G, resp. Milos Hudlicky

10

Co

Transformation of vinyl-type chlorides into ketones.
*O. Wiesner, Chem. Listy 37, 180-3 (1943); Chem. Zvest. 1944, 1, 348-9. Et bis(γ-chloroacetyl)malonate (I) (b.p. 104-1°) (2.063 g.) with 2 cc. concd. H₂SO₄ rapidly evolves HCl, which arises from the MeCCl-CHCl₂ group; the product of this reaction is di-*Et* 6-methyl-3-acetyl-3-cyclohexene-1,1-dicarboxylate, CH₃.CAc.CMc.*

CH₃.CH₂.C(CO₂Et)₂, b.p. 191-2°, b.m. about 310°; semi-carbazone, m. 143-8°. The ester is stable to HCl but alk. hydrolysis yields a dicarboxylic acid which on distn. in vacuo loses CO₂ rapidly, yielding 6-methyl-3-acetyl-3-cyclohexene-1-carboxylic acid, b.p. 200°, m. 97°. I, hydrolyzed with alc. NaOH, the EtOH removed with steam, and the residue acidified with H₂SO₄ and crystd. from C₆H₆, gives the dicarboxylic acid, m. 94°; decarboxylation yields 5-chloro-6-hexene-1-carboxylic acid (II), b.p. 121-2°, m. about 80°. Addn. of 19 g. of molten II to 50 g. concd. H₂SO₄ gives 8-hexanoic-1-carboxylic acid (acetylbutyric acid) (III), b.p. 144-5°; the evolution of HCl is 97% complete after 24 hrs.; the reaction mixt. is poured onto ice, the H₂SO₄ is removed with Ba(OH)₂, the filtrate is evapd. to a thick sirup and the III is distd. Addn. of a small amt. of H₂O to III gives a hydrate, m. 31-6°; this yields an oxime, m. 104°.

C. J. West

ASAC-ELA METALLURGICAL LITERATURE CLASSIFICATION

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	00
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3-Chlorocrotyl derivatives of barbituric acid. O. Wichterle and O. Némethy: *Chem. Listy* 57, 100-9 (1963).—By the reaction of MeCCl:CHCl:Cl (I) with compds. contg. acidic H, the radical 3-chlorocrotyl is attached to org. compds. *Ph* (3-chlorocrotyl)malonate (Ia): 260 g. I was added portionwise to 45 g. Na in 700 ml. EtOH and 320 g. $\text{CH}_3(\text{CO})\text{Et}$, the mixt. refluxed until EtOH had (ind.) off, water added, the mixt. refluxed until fractionated, giving 310 g. Ia, b.p. 157° , d_{20}^{20} 1.1015, n_D^{20} 1.4984, n_D^{25} 1.4831, n_D^{30} 1.4690, n_D^{35} 1.4644. *Et bis*(3-chlorocrotyl)malonate (II): 120 g. I was added to

$\text{NaCH}(\text{CO}_2\text{Et})_2$ from 23 g. Na in 450 ml. EtOH and 180 g. $\text{CH}_3\text{CO}_2\text{Et}$, and when the reaction ceased, the same amt. of NaOEt in EtOH was added, followed by 126 g. I, and the product worked up as above, giving 280 g. 74.5% II, b.p. 173–5°, d_4^{20} 1.1368, n_D^{20} 1.47493, n_D^{25} 1.47787, n_D^{30} 1.48227, n_D^{35} 1.49147. Diethylsuccinic acid, m. 144° was prepd. from 60 g. II heated with 17 g. Na and 240 ml. abs. EtOH 15 hrs. to 200°. No acetylenic deriv. of malonic acid was isolated. (3-Chloroacetyl)succinic acid (III): NaOEt from 60 g. Na and 805 ml. EtOH. 262 g. I, and 95 g. urea were refluxed 8 hrs. and the crystals decomp. with 290 ml. HCl and ice, giving 224 g. (98.6%) III, m. 182° (from water). Bis(3-chloroacetyl)succinic

acid: 50 g. II, NaOEt from 10 g. Na and 200 ml. EtOH, and 12 g. urea were refluxed 7 hrs. On acidification the mixt. gave 2 isomers, m. 104° (from 50% AcOH), and 178° (from 50% AcOH); the same mixt. was prep'd. by agitating 12.5 g. I with 4.4 g. barbituric acid and 100 ml. *N*-NaOH. Methyl-(3-chloroacetyl)barbituric acid (IV): to 38 g. methylbarbituric acid in 200 ml. *N*-NaOH was added 30.3 g. I, the mixt. stirred 10 hrs. at 60°, and the brown oil sepd., dissolved in NaOH, and decolorized with charcoal; acidification with concd. AcOH gave 31 g. (51%) IV, mp. 192° [from EtOH-H₂O (2:1) after 4 crystals]; IV was also prep'd. from Me(MeCCl:CHCl):C(CO₂Et)₂, b.p. 141-3°, obtained from NaCMe(CO₂Et)₂ [from 34 g. MeCl(CH₂Cl)₂], 4.5 g. Na, and 60 ml. EtOH] and 24.4 g. I in the usual manner; the condensation to IV was carried out with 48 g. ester, 8.5 g. Na, 110 ml. EtOH, and 15 g. urea. The following analogs, R(MeCCl:CHCl):C.CO.NH.CO.NH.CO, of IV are described: R = Et.

m. 150° (from water), obtained in 73% yield from 34.5 g. ethylbarbituric acid in 250 ml. *N* NaOH, 31.2 g. I, 0.5 g. CuSO_4 , and 1 g. KBr vigorously stirred 12 hrs. at 50°, or

from $\text{Et}(\text{MeCCl:CHCH}_2)_2\text{C}(\text{CO}_2\text{Et})_2$, b_p 155-60°, and urea in the way described above; *iso-Pr*, m . 100° (from 50% AcOH) (40 g. from 92 g. isopropylbarbituric acid in 540 ml. *N* NaOH, 2 g. KBr, 1 g. CuSO_4 , and 76 g. I stirred 30 hrs. at room temp., or from $\text{Me}_2\text{CH}(\text{MeCCl:CH-CH}_2)_2\text{C}(\text{CO}_2\text{Et})_2$); *Bz*, m . 151° (from 50% *N* NaOH, 2 g. from 85 g. butylbarbituric acid in 460 ml. *N* NaOH, 2 g. KBr, and 1 g. CuSO_4 , stirred at 40°, 75.6 g. I added during 1 hr., and the mixt. heated 7 hrs.; *cyclohexyl*, m . 201° (from 50% AcOH), *prepd.* from 100 g. cyclohexylbarbituric acid and 76 g. I, the crude product being dissolved in NaOH, decolorized, and *pptd.* with AcOH; *benzyl*, m . 170° (from *dist. EtOH*), *prepd.* from 5 g. benzylbarbituric acid in 22.8 ml. *N* NaOH, refluxed with 3 g. I and 3 ml. EtOH 3 hrs.; *cyclohexenyl*, m . 197° (from 50% AcOH), *prepd.* by refluxing 14 g. Na, 200 ml. EtOH, 18 g. urea, and 70 g. Et α -(3-chlorocrotyl)- α -cyanocyclohexeneacetate (V) 10 hrs., acidifying with HCl, hydrolyzing the *cryst. imino deriv.* by refluxing with *concd.* HCl, and purifying the product through the Na salt [V, b_p 181°, was obtained in 70-g. yield from 9.5 g. Na in 150 ml. EtOH, 80 g. Et α -cyanocyclohexeneacetate, and 58 g. I]. *S-Isopropyl(3-chlorocrotyl)barbituric acid*, m . above 250° (decompn.), was obtained in 100% yield from 10 g. isopropylthiobarbituric acid in 54 ml. *N* NaOH stirred with 7 g. I. *5,5-Diethyl-1-(3-chlorocrotyl)barbituric acid*, m . 93° (from 50% AcOH), from 4 g. veronal in 21.8 ml. *N* NaOH and 10 ml. EtOH stirred with 3 g. I 10 hrs. at 70°, and the oil purified by dissolving in NaOH and acidifying with AcOH.

Milton Hudlicky

CA

7

Essential features of cycle-acycle tautomerism. O. Wichterle. *Chem. Listy* 40, 28-32(1946).—A new classification of tautomerism is suggested: (1) equil. of 2 isolable individuals (keto-enol), (2) true tautomerism in which individual mols. with different structures cannot be sepd. into different individuals because of intermol. H bonds, and (3) mesomerism of mols. forming 6-membered rings by means of intramol. H bonds in which all mols. are identical. The so-called oxo-cycle tautomerism of sugars, sugar derivs., and some alkaloids belong to the 2nd class, since it is impossible to isolate pure individuals that would correspond to either formula.

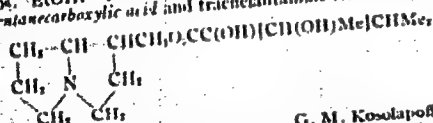
M. Hudlický

117 AND 118, ORDER		PROPERTIES INDEX	
<p>Syntheses of isobutylamines and acetylcholine. O. Wichterle and M. Hudlický (Lab. recherches chimiques maison Buta, Zlin). <i>Collection Czech. Chem. Commun.</i> 12, 120-37 (1947) (in French); cf. <i>C.A.</i> 41, 4148i. p-MeC₆H₄SO₂NH₂ (57 g.), heated 1 hr. at 170° with 20 g. 40% NaOH and an excess of MeCCl:CHCl₂ (I) with removal of H₂O by distn., then 2 hrs. with 30 g. more NaOH, yielded <i>N,N</i>-bis(3-chloro-2-butenyl)-<i>p</i>-toluenesulfonamide (II), b.p. 244°, m. 72-3° (from petr. ether). II (15 g.) with 4.5 g. (NH₄OH), H₂SO₄ for 2 days, followed by treatment with H₂O yielded the 4-methyl-3-acetyl-3-piperidine of <i>p</i>-toluenesulfonic acid [1-(<i>p</i>-tolylsulfonyl)-3-acetyl-4-methyl-1,2,5,6-tetrahydropyridine] (III), m. 101-2° (from 70% alc.) (semicarbazone m. 190-1° (decompn.)). An attempt to prep. III from II with concd. H₂SO₄ gave a tar. Attempted prepn. of <i>N,N</i>-bis(3-chloro-2-butenyl)-benzamide (IV) from I and Na benzamide gave only the mono deriv. (V). Attempted prepn. of the Na deriv. of V from V and NaNH₂, NaOEt, and Na in NH₃ failed. IV, m. 80-1°, was prepd. in 85.8% yield from (MeCCl:CHCl₂)₂NH.HCl and BaCl in NaOH soln. IV (46 g.) with 50 cc. H₂SO₄ yielded 4 g. of 4-methyl-3-acetyl-3-piperidine of benzoic acid [1-benzoyl-3-acetyl-4-methyl-1,2,5,6-tetrahydropyridine] (VI), m. 93-4° (from Et₂O) (semicarbazone m. 210-12° (decompn.)). VI (1.8 g.), refluxed with 16 cc. 20% HCl, yielded a syrup which, made alk., gave 4-methyl-3-acetyl-1,2,5,6-tetrahydropyridine (VII) (picrate m. 124.5°; reaction with semicarbazide gave a product m. 185°, contg. 2 mols. semicarbazide, 2 mols. H₂O, and 1 mol. VII.HCl). During the formation of VI, AcOH was produced; the mixt., after removal of sulfate, evapor. to dryness, and hydrolysis with 20% HCl, yielded a coump. C₁₀H₁₅NO₄, m. 300-3° (decompn.). I (1250 g.) with 2 l. NH₄OH yielded 548 g. (60.3%) <i>tris</i>(3-chloro-2-butenyl)-amine (VIII), b₁ 130°, b₂ 170°, b₃ 173-4°, n_D²⁰ 1.50788, n_D²⁵ 1.51120, n_D³⁰ 1.52022, n_D³⁵ 1.52883, d₄²⁰ 1.1234 (HCl salt, m. 183-4°). <i>Methyltris</i>(3-chloro-2-butenyl)ammonium chloride (as a by-product in the prepn. of (MeCCl:CHCl₂)₂NMe, m. 108° (from Me₂CO)); the isolate was obtained from VIII and MeI. VIII and I, let stand 2 months, yielded <i>tetrakis</i>(3-chloro-2-butenyl)ammonium chloride, m. 110-11° (from Me₂CO). R. W. S.</p>		<p>10</p>	

1ST AND 2ND CODES		3RD AND 4TH CODES	
PROCESSES AND PROPERTIES INDEX			
CA			
10			
<p>Dihydrobenzaldehyde and dihydrocinnamaldehyde. O. Wichterle and M. Hudlický. <i>Collection Czechoslov. Chem. Commun.</i> 12, 872-80(1947) (in French).—2-Acetyltetrahydrobenzaldehyde (27 g.) was heated with 9 g. NaOAc; repeated fractionation yielded 31% dihydrobenzaldehyde (I), b_p 88°, d_4^{20} 1.0176, n_D^{20} 1.53660; semicarbazone, m. 211-12° (decomp.); phenylhydrazones, m. 120°. From 6-acetyltetrahydro-α-tolualdehyde and NaOAc, heated at about 200°, was obtained 15% dihydro-α-tolualdehyde (II), b_p 84°. The major reaction product of $AcOCH:CHCH:CH_2$ and $MeCH:CHCHO$ (III) heated 5 hrs. at 180° was II, b_p 87.5°, d_4^{20} 1.0176, n_D^{20} 1.53660; and 99 g. NaOAc repeated 3 hrs. was obtained II, b_p 80-83°, n_D^{20} 1.53010, d_4^{20} 0.9846; semicarbazone m. 207°; phenylhydrazones m. 82-4° (from alc.). I and maleic anhydride resulted in 1-formylbicyclo-[2.2.2]-8-octene-2,3-dicarboxylic anhydride (IV), m. 133°; semicarbazone m. 201-8° (decomp.). II and maleic anhydride gave the 7-Me deriv. of IV, m. 108-9° M. Q. Webb</p>			
ASB-SLA METALLURGICAL LITERATURE CLASSIFICATION			
FROM SYMBOL		FROM SYMBOL	
GROUP		GROUP	

1-1 AND 1-2 COLUMNS		PROCESSES AND PROPERTIES USED		100 AND 10M COLUMNS	
<p><i>C₇</i></p> <p>Colorimetric analysis of oximes, ketones, and hydroxylamines. O. Wichterle and M. Hudlicky (Inst. recherches établissement, Bata & Zlin). <i>Collection Czechoslov. Chem. Commun.</i> 12, 661-71 (1947) (in French).—The reaction of Cl on ketoximes to form intensely blue chloro nitroso derivs., R₂C(Cl)NO, is converted to an analytical procedure. The aldokine derivs., however, rearrange to colorless RC(Cl); NOH. 1-Chloro-1-nitrosocyclohexane (I), obtained in 80-85% yield from 565 g. cyclohexanone oxime obtained in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (II) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (III) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (IV) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (V) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (VI) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (VII) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (VIII) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (IX) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (X) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (XI) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (XII) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (XIII) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (XIV) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (XV) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (XVI) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (XVII) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (XVIII) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (XIX) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (XX) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (XXI) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (XXII) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (XXIII) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (XXIV) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (XXV) in 1000 cc. H₂O and 375 g. 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Cl at 0° 1 hr. and steam (LXXVI) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (LXXVII) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (LXXVIII) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (LXXIX) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (LXXX) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (LXXXI) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (LXXXII) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (LXXXIII) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (LXXXIV) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (LXXXV) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (LXXXVI) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (LXXXVII) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (LXXXVIII) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (LXXXIX) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (LXXXX) in 1000 cc. H₂O and 375 g. Cl at 0° 1 hr. and steam (LXXXXI) in 1000 cc. H₂O and 375 g. 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Alkaloids of Trachelanthus korolkovi. IV. Structure
of trachelantamine. G. P. Men'shikov (Acad. Med.
Sci., Moscow). J. Gen. Chem. (U.S.S.R.) 17, 343-6
(1917); cf. C.A. 4, 1932b.—Hydrolysis of trachelan-
tamine gives the previously characterized and identified tri-
amine gives the previously characterized and identified tri-
amine gives the previously characterized and identified tri-
amine gives the previously characterized and identified tri-
 $\text{C}_{10}\text{H}_{18}(\text{OH})_2\text{CO}_2$, whose structure is shown to be $\text{C}_{10}\text{H}_{18}(\text{OH})_2\text{CO}_2$.
Reduction of 20 g. I by boiling with addn. of 100 cc. H_2 and
and 4 g. red P 8 hrs., followed by addn. of the distillate being exd.
4 g. red P and steam distl., with the distillate being exd.
with EtO and the evap. ext. boiled with 15 g. Zn and
with 15% HCl, followed by steam distn., concn. of the
distillate after treatment with CaCO_3 and acidification
by 10% HCl gave 2.7 g. dihydroxypropionic acid, b.p.
202–6°; chloride, mp. 115.5–16.5° (from petr. ether). Oxida-
tion of 15 g. I by heating with Hg oxide (from 50 g. Hg)
in 400 cc. H_2O gave a volatile yellow-green oil (M_r 116,
identified as $\text{Me}_2\text{CHCOCOMe}$; ozonome,
mp. 116–17° (from abs. EtOH), by heating 1 g. of the oil
ketone with 2.5 g. PINHNH_2 in 4 cc. AcOH and 4 cc.
abs. EtOH. Therefore, I is 3-methyl-3,4-dihydroxy-
pentane-carboxylic acid and trachelantamine is:



G. M. Kosolapoff

CH₂, CH₂ G. M. Kossolapov
Addition of nitroso compounds to a conjugated system. Collection
(O. Wichterle, *Chém. Commun.* 12, 292-301 (1947). The
Cercothol. Chem. Commun. 12, 292-301 (1947). The
electronic structures of the C in carbon monoxide and the
N in nitroso compds are similar to that of S in SO₂ which
combines with dienes to form cyclic sulfones with a penta-
gonal ring. The reaction of PANO (I) or 2-chloro-2-

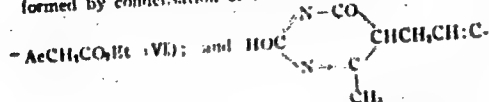
nitrosopropene (II) or 1-chloro-1-nitrosochloropropane (III) with 1,3-butadiene (IV) or derive, did not give the expected cyclic amine oxide but H₂O-insol. products of a 3,4-mixopyrrolidine structure. A mixt. of I and IV reacted so violently that diin. with CaH₂ was used. A soln. of 25 g. II and 60 g. I in 350 cc. CCl₄ changed from green to brown in 12 hrs. and a slight yellow cryst. ppt. formed. Removal of the solvent and steam distn. gave 63 g. 1-phenyl-3,4-oxido-pyrrolidine (V), as white lustrous flakes, m. 48° (from 70% EtOH). Distn. of V with Zn gave 1-phenylpyrrole. V (16.1 g.) in 50 g. MeOH absorbed in 3 hrs. 2240 cording to Adams at 1000 mm. giving a clear vol. c. II (at 0° and 799 mm.). (= theory) to give a clear vol. low oil, C₁₀H₁₁NO, b. 105–7°, d₄ 1.0017, n_D 1.6536, mol. refraction 49.28. Only 0.006% active II at most was found by the Zerewitinoff method. Refluxing 20 g. V in 85 cc. by the Zerewitinoff method. Refluxing 20 g. NaOH soln., HOAc and 30 g. Zn 30 min., then adding aq. NaOH soln., gave a yellow oil. Distn. gave a 1-phenylpyrrolic fragrance VI, b. 115–50°, m. 101–2° (from MeOH), and 7 g. tion (VI), b. 115–50°, m. 101–2°. VII, distd. with H₂, 2-acetoxypyrrolidine (VII), b. 164°. VII, distd. and then PO, (c. 1.71), gave a little HOAc in the distillate and then VI. VI, hydrogenated in 95% EtOH over PtO₂ gave a product contg. 0.005% N (theory for 1-phenylpyrrolidine 0.53%). I (4.3 g.) and 3.4 g. CH₃CMeCMe:CH₂ in 70 cc. CCl₄ gave 2.6 g. 1-phenyl-3,4-dimethyl-3,4-oxido-pyrrolidine, m. 39–40° (white crystals from 90% EtOH). 1-Phenylbutadiene (3.1 g.) and 2.6 g. I in 20 hrs. gave 2.6 g. 1,2-diphenyl-3,4-oxido-pyrrolidine, light yellow crystals, m. 85° (from EtOH). 3,4-Oxido-pyrrolidinium crystals, m. 85°, prep'd. in 3.5-g. yield from 6 g. II (from chloride (VIII), prep'd. in 25 cc. of 2.5 M IV in the chlorination of Me:C:NH), 25 cc. of 2.5 M IV in the chlorination of EtOH in 6 hrs., m. 132° (long needles C₁₀H₁₁N), and 3.5 cc. EtOH in 6 hrs., m. 132° (long needles C₁₀H₁₁N). VIII will not crystallize and yields are low from EtOH. VIII will present in the reaction mixt. VIII can also be obtained in 35-g. yield from 68 g. III and 200 cc. of

2,3 M IV in C_6H_6 : 3,4-Oxidopyrrolidine (IX), obtained
from VIII by KOH, bp 61-2°, 3,4-oxido-1-pyrrolidinecarbox-
anilide, obtained from 0.42 g. IX and 0.6 g. $PhNCO$ in 10
cc. Et_2O , m. 81-2° (from $EtOH$). 3,4-Dimethyl-3,4-
oxidopyrrolidine, obtained in 1-g. yield from 4.1 g. 2,3-
 CH_3 : $CMcCMc$: CH_3 and a 1.2 M II in C_6H_6 refluxed 2
hrs., bp 63-5°; picrate m. 180° (from $EtOH$).
John W. Green

CA

(Chloroacetyl)isethiourea. J. Procházka and O. Wichterle. *Collections Czechoslov. Chem. Commun.* 14, 156-61 (1949) (in French).—Three new derivatives of $\text{MeCCl:CH:CH}_2\text{SC(NH}_2\text{)}_2$ (I) are reported: $\text{[MeCCl:CH:CH}_2\text{SC(NH}_2\text{)}_2\text{]}^+\text{Cl}^-$ (II), formed by condensation of I with thiourea $\text{NH}_2\text{C(=S)NH}_2$ (III); $\text{MeCCl:CH:CH}_2\text{SC:N.CO.CH}_2\text{CMe}_2\text{N}$ (IV),

formed by condensation of the free base (V) of II with



(CH₃)₂CH (VII), formed from II and (VIII), the chloroacetyl deriv. of VI. I (300 g.) and 304 g. III in EtOH refluxed for 1.5 hrs., filtered, and let stand formed beautiful crystals of II, m. 141-2° (from EtOH-Me₂CO or 1:3 HCl) or 141.5-5.5° (from NaCl soln.), sol. in H₂O, EtOH, and Me₂CO. II (220 g.) in 2 l. H₂O was converted by a 15% soln. of NaOH or aq. NH₄OH to pale pink scaly crystals of V, m. 82° (from C₆H₆). Heat decoupled V into MeC-CH:CHCH₂SH (IX), b. 47-8°, b.p. 145°, also formed by dropwise addn. of 10% of a soln. of NaSH to I, and identified by its 2,4-(OHN)C₆H₃Cl₃ deriv., m. 92° (from EtOH). After long standing, 70% IV, m. 203-3.5° (from EtOH), was obtained as its monohydrate either by dropwise addn.

of 25 g. KOH in 20 cc. H₂O to a cooled mixt. of II in 40 cc. H₂O and 32.6 g. VI, or by addn. of 27.7 g. VI to 35 g. V in 160 cc. hot EtOH. VII, m. 224-5° and 237-8° (after many recrystns. from dil. EtOH), was prepd. either from 28 g. VIII with 36.6 g. II in 30 cc. H₂O and 9.7 g. NaOH in 10 cc. H₂O, or from 178 g. II and 194 g. VIII in 220 cc. hot denatured EtOH and 50 g. NaOH in 50 cc. dist. H₂O added to the cold soln. The treatment of I with NH₄CNS and with concd. H₂SO₄ is described. Equimolar quantities of I and NH₄CNS (or KCNS) stirred in cold alc. or on a water bath yielded MeCCl:CH:CH₂SCN (X), b. 103-5°. An attempt to transpose X by distg. it at water-pump pressure failed for it formed an explosive mixt. X b. 97-130° formed a product, b. 167-9°, which was slightly different from X and which did not burn the skin, but with NH₄ gave NH₄Cl. A product, C₁₁H₁₂N₂S₂O₄ (XI), of unknown structure, with mol. wt. 288.2 (theoretical), m. 241°, was formed by removing 98% of the theoretical amt. of HCl from 350 g. II by vigorously shaking it while adding 65 cc. concd. H₂SO₄; after removal of the HCl by dil. alkali and washing the soln., there formed crystals, m. 231-2° (from H₂O), which were insol. in common org. solvents, but when recrystd. from H₂O to a common org. solvent, the solid became sol. in H₂O. Refluxing for 25 hrs. caused complete soln., giving a sirup which formed crystals m. 241° when mixed with H₂O.

Helen L. Whidden

CA

10

Addition of nitroso compounds to conjugated systems.
 O. Wichterle and J. Vogel. *Collection Czechoslov. Chem. Commun.* **16**, 209-18 (1949) (in English); cf. C.I. **42**, 3511. — Present work confirms the results of Arbutov (C.I. **43**, 830c) that the reaction of PhNO and $\text{Me}_3\text{C}-\text{CH}=\text{CH}-\text{NO}$ (I) gives 2-phenyl-3,6-dihydro-1,2-naphthoquinone (II) rather than 1-phenyl-3,4-oxido-pyrroline. The action of various Grignard reagents on I results in the splitting of the O—N bond and hydrogenation to $\text{PhNHCH}_2\text{CH}=\text{CH}-\text{CH}_2\text{OH}$ (III), b. 156–60°; the alkyl radicals formed from the Grignard either join or disproportionate into an olefin and a para-substituted phenol. Dehydration of II by H_3PO_4 gives 1-phenyl-3-pyrroline (IV); methiodide, m. 138° (from EtOH). Ozonization of III and subsequent hydrogenation of the ozonide with PtO_2 gives 4-phenylmorpholine, m. 54.3–9°. EtMgBr (IV) and 2-phenyltetrahydro-1,2-naphthoquinone give 81°; 4-phenylamino-2-butanol (V), b. 157°, n_D²⁰ 1.5629, d₄²⁰ 1.0508, which yields an acid oxalate, white needles from MeOH, m. 124.8–5° (decomp.); hydrogenation of II also yields V. Dehydration of V by H_3PO_4 gives 1-phenylpyrrolidine; picrate, m. 115–15.5°. IV did not react with either $\text{PhNMeOCH}_2\text{CH}=\text{CH}_2$ or PhNMeOPr .
 P. M. Downey

CA

The addition of nitro compounds to diones. III, 3,6-dihydro-1,2,2H-oxazine. (O. Wichterle and J. Novák. Collection Czechoslov. Chem. Commun. 15, 300-211 (1950) (in French); cf. C.A. 44, 1515c. The Zn and catalytic hydrogenation of $O_2CH_2CH=CHCH_2NR$ (I) is described. In the reduction of I, HCl (R = Et) with Zn and AcOH, the ring is split to give 1-amino-2-buten-1-ol (II). With PtO_2 , 3 products are obtained, the tetrahydro-1,2,2H-oxazine (III), pyrrolidine (IIIA), and 1-amino-2-butan-1-ol (IV). IV is also obtained by the Zn and AcOH reduction of III. I (R = Et) (5 g.) in 30 cc. Ac_2O is stirred with 5 g. Zn dust, and after the vigorous reaction subsides, the Zn salt is removed in vacuo, the residue, after purification, II as a viscous liquid, b.p. 84-5°, n_D²⁰ 1.4992, d₄ 1.011. I (60.5 g.) in 250 cc. MeOH is hydrogenated in the presence of PtO_2 catalyst, the solvent removed, the residue treated in 100 cc. H_2O with 60 g. KOH, and the basic ppt. extd. with ether and fractionally distd., giving IIIA, b.p. 74-6° (identified as the chloroplatinate, m. 211°), III, b.p. 70-1°, d₄ 0.9010, n_D²⁰ 1.4064 (picrate, m. 151-2°), and IV, b.p. 70-2°. III and PhNCS (methyl 3-oxide, m. 60°). IV and PhNCS give 1-(1-hydroxybutyl)-3-

phenyl-2-thioxoate, m. 105°. I (10 g.) reduced with 1.5 g. LiAlH₄ gives 2-ethyl-3,6-dihydro-1,2,2H-oxazine (VI), b.p. 84-5°, n_D²⁰ 1.4260, d₄ 0.9071; picrate, m. 80°. V (5 g.) with 0.5 g. Zn in 30 cc. AcOH gives 3.2 g. 1-ethylamino-2-buten-1-ol, b.p. 82-3°, n_D²⁰ 1.4882, d₄ 0.9170. III (10 g.) and 13 g. EtNH₂ give 2-ethyltetrahydro-1,2,2H-oxazine (VII), b.p. 61-5°, n_D²⁰ 1.4372, d₄ 0.8974. VI (5 g.) with Zn gives 3.1 g. 1-ethylamino-2-butan-1-ol, b.p. 70°, n_D²⁰ 1.4518, d₄ 0.9121. When 10 g. I in 25 cc. H_2O acidified with HCl is treated with 6 g. $NaNO_2$ in the cold, the soln. turns yellow and deposits only yellow-brown drops; the oil and ether exts. of the aq. layer are combined, dried, and distd. after removal of the solvent, giving 2-nitroso-3,6-dihydro-1,2,2H-oxazine (VIII), b.p. 71°. VII (5 g.) with 5 g. Zn dust at 15° gives N and impure material, b.p. 50-105°. III (10 g.), similarly nitrosated, gives 10.2 g. 2-nitrosotetrahydro-1,2,2H-oxazine, b.p. 75-6°. On reduction of the latter with Zn only BuOH could be isolated. From 10 g. I in 50 cc. MeOH and 30 g. MeI is obtained 7.2 g. 2,2-dimethyl-3,6-dihydro-1,2,2H-oxazinanium iodide (VIII), m. 133° (decolor.). VIII with Ag_2O or $AgOAc$ gives the unstable cationary hydronide and acetate. The following are also prepd.: 2-Bz, b.p. 153-5°; 2-Ac, b.p. 83-4°; 2-benzyl, m. 78°. Derivs. of III: 2-Bz, b.p. 152-3°; 2-Ac, b.p. 81-4°. An attempt to prep. the 2-formyl deriv. of I was unsuccessful. I with HNO_3 gave an explosive product. Bernard Klein

1951

General Physical
Chemistry 2

cf

Bockmann rearrangement of cyclohexanone oxime.
Kinetics of final stages of the reaction. I. Oto Wichterle
and Jan Rofek (Tech. Univ., Prague, Czech.). *Chem.
Listy* 45, 257-9 (1951).—Because the reaction is exothermic
and rapid, the kinetic studies of the Bockmann rearrange-
ment offer many difficulties. However, the final stages of
the reaction can be easily followed, since the oxime is dissol-
ved with the product of rearrangement which lowers the temp.
peak from the heat developed in the reaction. The measure-
ments were carried out in 30-80% solns. of cyclohexanone
oxime and ϵ -caprolactam in 5% oleum. The reaction is of
the 1st-order. The velocity consts. at 10-80° were detd.
II. *Ibid.* 379-80.—The effect of the concn. of SO_3 in
oleum on the reaction rate of the Bockmann rearrangement
of cyclohexanone oxime was followed at const. temp. with a
const. amt. of oleum. Changes of the SO_3 content in
approx. 100% H_2SO_4 influenced the reaction rate most
effectively. The min. effect was with a change of concn. of
 SO_3 in 5-10% oleum. The reaction was 1st-order, and its
rate const. at 20° was $1.15-0.2 \times 10^{-4}$, depending on the
 SO_3 content in the oleum. M. Hladický

CA

Kinetics of the Grignard reaction in the case of sterically hindered esters. O. Wichterle and P. Haterka. *Collection Czechoslov. Chem. Commun.* 15, 1021-3 (1951) (in German). —Triebs (*C.A.* 40, 5396) has found that while the Grignard reaction is instantaneous with most carbonyl compds., it can be followed in the case of carboxyl esters. Accordingly

the reactivity of various esters with MeMgI has been detd. as an aid to configuration detas. To eliminate mutual interference by steric hindrance of the acid and the alc. radical, Me esters of various acids and acetates of alc. were investigated. The results in general are in agreement with those of Newman (*C.A.* 43, 4644e), who showed that the greater hindrance is found in the esters that have a large no. of atoms in position 8 with regard to the carbonyl and a somewhat weaker hindrance in position 6. Exceptions were found in the case of the abnormally great reactivity of iso-BuOAc and the small reactivity of EtOAc. The rule does not hold with alkoxy esters. Alfred Hoffman

WICHTERLE, OTTO

Wichterle, Otto. Anorganicka chemie. (1. vyd.) Praha, Nakl. Ceskoslovenske akademie ved, 1953. 521 p. (Ceskoslovenska akademie ved. Veda vses. Sekce chemicka, sv. 8) (Inorganic chemistry. 1st ed. bibl., illus., indexes, tables)

SO: Monthly List of East European Accessions, (SEAL), LC, Vol. 4, No. 11, Nov. 1955, Uncl.

WICHTERLE, OTO.

1 of 2

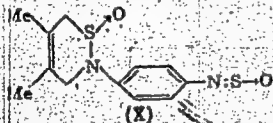
Addition of dienes to thionylamines. Preparation of heterocyclic compounds of 1,2-thiazine series. Oto Wichterle and Jan Ritzel (Vysoká škola chem., Prague, Czech.). Chem. Zvesti 47, 1788-86 (1953); Collection Czechoslov. Chem. Commun. 19, 132-98 (1954) (in Russian); cf. C.A. 42, 5561; following abstr. — A new type of the Diels-Alder reaction was discovered in the addn. of dienes, $\text{CH}_2=\text{CHCH}=\text{CH}_2$ (I) and $\text{CH}_2=\text{CHCM}=\text{CH}_2$ (II), to aromatic thionylamines

($\text{RN:S}=\text{O}$) which led to substituted 2,3-dihydro-6H-1,2-thiazine 1-oxides (III), a new type of heterocycle. The reactions of III were studied for 4,5-dimethyl-2-phenyl-2,3-dihydro-6H-1,2-thiazine 1-oxide (IIIa) which gave on acidic hydrolysis $\text{PhNHCH}_2\text{CHMeCM}=\text{CH}_2$ (IV), on alk. hydrolysis 1-phenyl-3,4-dimethylpyrrole (V) (see following abstr.), by LiAlH_4 reduction 2-phenyl-4,5-dimethyl-2,3-dihydro-1,2-thiazine (VI), and by oxidation IIIa 1,1-dioxide (VII), the 4,5-epoxy deriv. (VIII) of IIIa, and the 4,5-epoxy deriv. (IX) of VII. The addn. does not occur with aliphatic thionylamines. The thionylamines were prepd. by heating amines with SOCl_2 on the steam bath; until no more HCl escaped, and distg. or crystg. the crude product. Conditions and properties of thionylamines are listed (lit. of amine, ml. SOCl_2 , ml. C_6H_6 , reaction time in hrs., b.p., m.p., % yield): $\text{PhN:S}=\text{O}$, 1020 ($\text{PhNH}_2, \text{HCl}$), 600, 2300, 15, b.p. 84-8°, —, 85; $p\text{-ClC}_6\text{H}_4\text{N:S}=\text{O}$, 17.5, 11, 160, 2, b.p. 123°, 55-8°, 90.5; $m\text{-ClC}_6\text{H}_4\text{N:S}=\text{O}$, 50, 30, 100, 4.5, b.p. 114.5°, 21°, 98.3; $o\text{-MeC}_6\text{H}_4\text{N:S}=\text{O}$, 39.8, 30, 100, 0.7, b.p. 129°, —, 90; $p\text{-MeOC}_6\text{H}_4\text{N:S}=\text{O}$, 21.8, 15, 160, 2, b.p. 132°, 63-4°, 80; $p\text{-MeOC}_6\text{H}_4\text{N:S}=\text{O}$, 12.5, 20, 100, 2.7, —, 21°, 93.8; $p\text{-ClC}_6\text{H}_4\text{N:S}=\text{O}$, 12.5, 20, 100, 2.7, —, 113.5-14.5°, 65. BuNH_2 (78 g.) in 100 ml. Et_2O was treated, at -10° , with 43 g. $\text{SOCl}_2, \text{HCl}$ filtered off, and the on the steam bath 1 hr., the $\text{BuNH}_2, \text{HCl}$ filtered off, and the residue, distd. yielding 12.2 g. (28.4%) $\text{BuN:S}=\text{O}$, b.p. 64-5°, d₂₀ 1.0165. The addn. was carried out by boiling a slight excess of the diene with the thionylamine several hrs. on the steam bath. Refluxing 70 g. $\text{PhN:S}=\text{O}$ and 45 g. II 8 hrs., and distg. the mixt. at 1-2 mm. gave 12 g. (17%) unreacted $\text{PhN:S}=\text{O}$ and a residue which yielded 80 g. (72%)

OTO WICHTERLE

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IIIa, m. 70.5-80.7° (from cyclohexane). M.ps. and % yields (based on the reacted thionylamines) of III (R = Me, R' given) were as follows: *m*-O₂NC₆H₄, 172-3°, 80; *p*-O₂N-C₆H₄, 173-5° (decompn.), 40; *o*-MeC₆H₄, 82.5-3.5°, 65; *p*-MeC₆H₄, 101.5-1.5°, 89; *p*-MeOC₆H₄, 110.5-11°, 88; *p*-MeOCC₆H₄, 106-7°, 87; *α*-C₆H₅, 128-30°, 70; *β*-C₆H₅, 153.9-4.3°, 64; *m*-ClC₆H₄, 136-7°, 70. The compds. were crystd. from C₆H₆-petr. ether, or from EtOH. I (29 ml.) and 29 ml. PhN:S=O heated in a sealed tube 8-16 hrs. at 100° gave after crystn. from C₆H₆: 2-phenyl-2,3-dihydro-6H-1,3-thiazine 1-oxide (IXa), m. 97-7.5° (from C₆H₆-petr. ether), and its mol. compd. with 1 mole PhNH₂, m. 74.2-5.2°. The same addn. compd., m. 74.2-6.3°, was prepd. in 95% yield by mixing 0.74 g. IXa and 0.36 g. PhNH₂ in 2.5 ml. C₆H₆. *p*-C₆H₄(N:S=O)₂ (6 g.) and 8 ml. II gave, after heating 11 hrs. on the steam bath, 4.3 g. unstable X, m. 157-82°, which hydrolyzed by 1 day standing to the *p*-H₂N analog of IIIa, m. 190-7° (decompn.). Heating 30 g. IIIa with 15 ml. HCl and 45 ml. H₂O 30 min. on the steam-



bath, alkalinizing the mixt. with 7 g. NaOH, extg. with

Et₂O and evap. the Et₂O ext. gave 23 g. (95.7%) IV, b_p 101°, d₄ 0.8194, d₁₅ 0.9442, n_D²⁰ 1.5380 (HCl salt, m. 120-32°; the ultramine, picrate, and *Be* derivatives are oils), hydrogenation over PtO₂ showed 0.94 double bond. When the hydrolysis was carried out with 10% H₃PO₄, a small amt. of PhNHCH₂CHMeCMc(OH)Me, b_p 135°, n_D²⁰ 1.5610, was obtained. IIIa (2.3 g.) boiled with 0.5 g. LiAlH₄ in 50 ml. Et₂O the mixt. decompd. with 3 ml. H₂O, and the Et₂O layer evapd. yielded 1.04 g. of an oil which gave VI, m. 93-4° (from MeOH). Treating 1.1 g. IIIa with 825 mg. Br₂O₃ in 25 ml. CHCl₃ 16 hrs. at -15° gave a good yield of VIII, m. 147-8° (from EtOH-H₂O). Similarly from 16.5 g. IIIa and 0.080 mole Br₂O₃ were obtained 5.7 g. VIII and, by chromatography, 1.4 g. VII. VII, m. 151.5-2.5°, was also prepd. (1.4 g., 40%) by treatment of 4.4 g. IIIa in 80 ml. EtOH with 16 ml. 5% NaOH and 16 ml. 20% H₂O₂ at room temp. IIIa (1.1 g., 0.005 mole) with 30 ml. CHCl₃ contg. 0.013 mole Br₂O₃ yielded, after 20 days at room temp., 1 g. (79%) IX, m. 81.7-2.7° (from C₆H₆-petr. ether). Heating 2.37 g. VIII with 10 ml. 5% HCl yielded a small amt. of a compd. m. 121°, and 1.24 g. (55%) of an oil distg. at 0.2-0.3 mm. at 111-28° (bath temp.), d₄ 1.0240, n_D²⁰ 1.5540, probably PhNHCH₂CHMeCMc(OH)Me. Heating 8.8 g. IXa with 5.5 ml. HCl and 15.5 ml. H₂O 40 min. on the steam bath, and 5 min. to the b.p., alkalinizing the mixt., extg. with Et₂O, and evap. the ext. gave 5.77 g. (87%) PhNH-CH₂CH₂CH₂CH₂ (XI), b_p 133°, d₄ 0.9578, n_D²⁰ 1.5538; acidic oxalate, m. 149.5-61° (decompn.) (from EtOH); HCl salt, m. 137.5-40.5° (decompn.). Oxidation of the Ac deriv. of XI by 163° gave CH₃CO. Treating 1.93 g. IXa with 0.11 mole Br₂O₃ in a mixt. of C₆H₆, CHCl₃, and CCl₄ gave 1.35 g. (65%) 2-phenyl-2,3-dihydro-6H-1,2-thiazine 1,1-dioxide, m. 85-6° (from MeOH and EtOH).

M. Hudlický

WICHTERLE, OTO

CZECH

Our technology of caprolactam. Oto Wichterle. Chem. Průmysl 4(29), 309-71(1954).—Some problems and experiences of the caprolactam production as worked out in Czechoslovakia are discussed. CCl_4 , originally used as a solvent of cyclohexanone (I) oxime (II) to enable removal of heat during the Beckmann rearrangement, was not useful because of its chlorinating tendency. The prepn. of H₂NOH proposed by Raschig as a batch process could be modified into continuous process and the 100% SO_2 was replaced by crude gas from the calcination of pyrite. In the existing modification the nitrite and sulfite soln. flows down on the inner surface of Pb-pipes cooled by brine, with the SO_2 gas blown in countercurrently. The prepn. of II from I and H₂NOH is a batch process. The Beckmann rearrangement is done continuously by introducing molten fresh oxime together with oleum into the reaction mixt. The study of kinetics of this reaction revealed the dependence of the reaction velocity on the concn. of SO_2 in the oleum. The velocity is practically 0 with H₂SO₄ of less than 100% concn. With the increasing concn. of SO_2 in oleum the reaction velocity increases sharply (diagram is given). However, a range exists between 5 and 12% SO_2 where the velocity is practically constant. This condition is found most advantageous for the stability of the process (cf. C.A. 46, 10800f).

L. A. Helwich

WICHTERLE, O.; ROCEK, J.

Addition of dienes to thionylamines; preparation of heterocyclic compounds of 1,2-thiazine series [with summary in German]. Sbor. Chekh. khim. rab. 19 no. 2: 282-297 Ap '54. (MLRA 7:6)

Plastic Melamine
1. Institut plastmass Prashakogo Khimicheskogo instituta.
(Thiazine)

WICHTELEK, O.; KOLINSKY, M.

"Addition of Chloroprene to Nitroso Compounds." p. 493, (COLLECTION OF CZECHOSLOVAK CHEMICAL COMMUNICATIONS. SBORNÍK CHEKOSLOVATSKÝCH KHEMICKÝCH PRÁV, Vol. 19, No. 3, June 1954, Praha, Czechoslovakia)

SO: Monthly List of East European Accessions, (EEAL), LC, Vol. 4
No. 5, May 1955, Uncl.

WICHTERLE, O.

Continuous processes. I. Nitration of cyclohexane.
O. Wichterle, M. Kollinsky, and S. Svastal (Vysoká škola
technická, Praha, Czech.). Chem. Listy 48, 81-83 (1954).
An app. for continuous nitration of cyclohexane is thor-
oughly described. The app. is a universal type of con-
tinuous autoclave enabling one or more liquids to be added
at a certain vol. rate which can be changed even during
the operation. Conversions up to 13.9% nitrocyclohexane
(based on cyclohexane) were obtained. M. Hudlický

WICHTERLE, Otto

CZECH

Acetylation of vinylidene chloride. Otto Wichterle and
 J. Vögel (Vysoká škola chem., Prag, Czechoslovakia). *Chem. Listy*
 48, 1225-31; Collection, Czechoslov. Chem. Commun. 19,
 1197-1204 (1954) (in English).—A new modification of the
 prepn. of CH_2Cl_2 (I) from CH_2Cl_2 (II) is described:
 II was added dropwise to the top of a column filled with
 granulated Zn. Water was boiled in a flask connected to
 the lower end of the column. The top of the column was
 fitted with a reflux condenser fed with 30° warm water.
 The condenser returned II and H_2O to the column while I
 passed to a condenser and receiver cooled with ice water;
 b.p. of I, 31-1.5°. Adding to a stirred mixt. of 50 g.
 AlCl_3 and 100 g. AlCl_3 73 g. I at 0 to -5°, decomp. the
 mixt. with ice, and steam distg. gave 83 g. $\text{MeCOCH}:\text{CCl}_2$
 (III), stable when wet and covered with H_2O , b. 153-0°
 d₄ 1.3008, n_D 1.4028. Dry III decomp. slowly, releasing
 HCl . III phenylhydrazones: m. 77-8° (semicarbazone).

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 6-ethyl-2-methyl-4-chloro-1H-pyran-3-one (IV), b. 230-231°, d. 1.180, n. 1.430 (from H₂O). IV is considered to be either 2-methyl-6-chloro-1H-pyran-3-one, or 6-methyl-4-chloro-1H-pyran-3-one, since its hydrogenation over PtO₂ in MeOH gave 62% Me(CH₂)₂CO₂Me, b. 160-2°. Adding the ester (1.7 g.) to a mixt. prepd. by the reaction of PhNH₂ (2.12 g.) with EtMgBr (from 2.84 g. EtBr and 0.43 g. Mg, 10% Me(CH₂)₂CONHPh, m. 60-7°. Treating 0.9 g. III with 5 g. concd. H₂SO₄ at 80-90° 2 hrs., removing the HCl liberated with a stream of air, raising the temp. after 1 hr. to 100°, adding 1 g. H₂SO₄, pouring the mixt. after 2 hrs. at 100° into H₂O (the product solidified), steam distg. 2 g. of III, dissolving the residue in Et₂O, and distg. the ext. in vacuo gave 37 g. of a compd., C₁₁H₁₃ClO₂, b. 143-144°, d. 1.180 (from MeOH), mol. wt. 208.2. Bromination of V in CCl₄ gave a compd., C₁₁H₁₃BrClO₂, m. 142-3°. Hydrogenation of 25 g. V in 200 ml. MeOH over 0.1 g. PtO₂ gave, after 20 hrs. (consumption 4.2 moles H₂) 7 g. EtMgCH(CH₂)₂CO₂Me (VI), b. 65.5-7°, d. 0.833, n. 1.418. Hydrolysis of VI gave 80% EtMgCH(CH₂)₂CO₂H (VII), b. 115°, d. 0.9105, n. 1.4300. Adding 4.95 g. VI to 0.6 g. 14% NaOH in Et₂O, refluxing the mixt. 1 hr., and decarboxylating the mixt. with 2% HCl gave 3.24 g. MeEtCH(CH₂)₂OH (VIII), b. 180-0°. Refluxing 3.24 g. VIII, 11.3 g. exo-tropic HBr, and 2.9 g. concd. H₂SO₄ 5 hrs. yielded 2.13 g. EtM-CH(CH₂)₂Br (IX), b. 185-9°. Treating 2.13 g. IX in Et₂O with 0.205 g. Mg, and decarboxylating the Grignard agent with H₂O gave 0.4 g. EtM-CH(CH₂)₂Me, b. 114-16°.

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The identity of VII was checked by comparison with a synthetic comp^d, prepd. as follows: MeCHBrEt (100.5 g.) and 80 g. CH₃(CO₂Et)₂ gave 70 g. MeEtCHCH(CO₂Et)₂, the hydrolysis of the ester and decarboxylation of the free acid yielding 85% EtMeCHCH₂CO₂H, b. 191-8°. Treatment with SOCl₂ and subsequent treatment with EtOH gave 93% EtMeCHCH₂CO₂Et, b. 156-9°, the reduction of which with Na in EtOH-PhMe yielded 44% EtMeCHCH₂CH₂OH, b. 151-4°. Transforming the alc. with PBr₃ at 10° to 70% EtMeCHCH₂CH₂Br, b. 145-8°, treating the bromide with Na and CH₃(CO₂Et)₂, hydrolyzing the ester and decarboxylating the free acid at 200° yielded 67% VII, b. 228-32°. *Ms. ester*, prepd. with CH₃N₃, b. 180-5°, anilide, m. 40-40.5°, identical with that prepd. from VI. M. Hudlický

WICHTERLE, O.

CZECH

Pneumatic valve for liquids. J. Pinkava and O. Wichterle (Vysoká škola chem., Prague). Chem. listy 48 (1954).--A pneumatic valve for liquids is described, the principal part of which is a glass float closing a ground glass valve with a glass ball. The flow of the liquid is regulated by gas pressure. The valve is suitable for feeding liquids against changing pressure, vacuum, or superatm. pressure or for feeding several liquids in const. ratio, and in relation to liquid pressure, amt. of flowing liquid, d., and viscosity. Accuracy is within $\pm 1.5\%$. M. Hudlický.

WICHTERLE, O.

Czechoslovakia

Annual meeting of the Chemical Society in the German Democratic Republic from 19-22 October 1955.

"Beitrag zur Kinetik der kationischen Polymerisation von Isobutylen"

SO: Chemische Technik, Feb 1956, Unclassified.

WichTeELE, O To

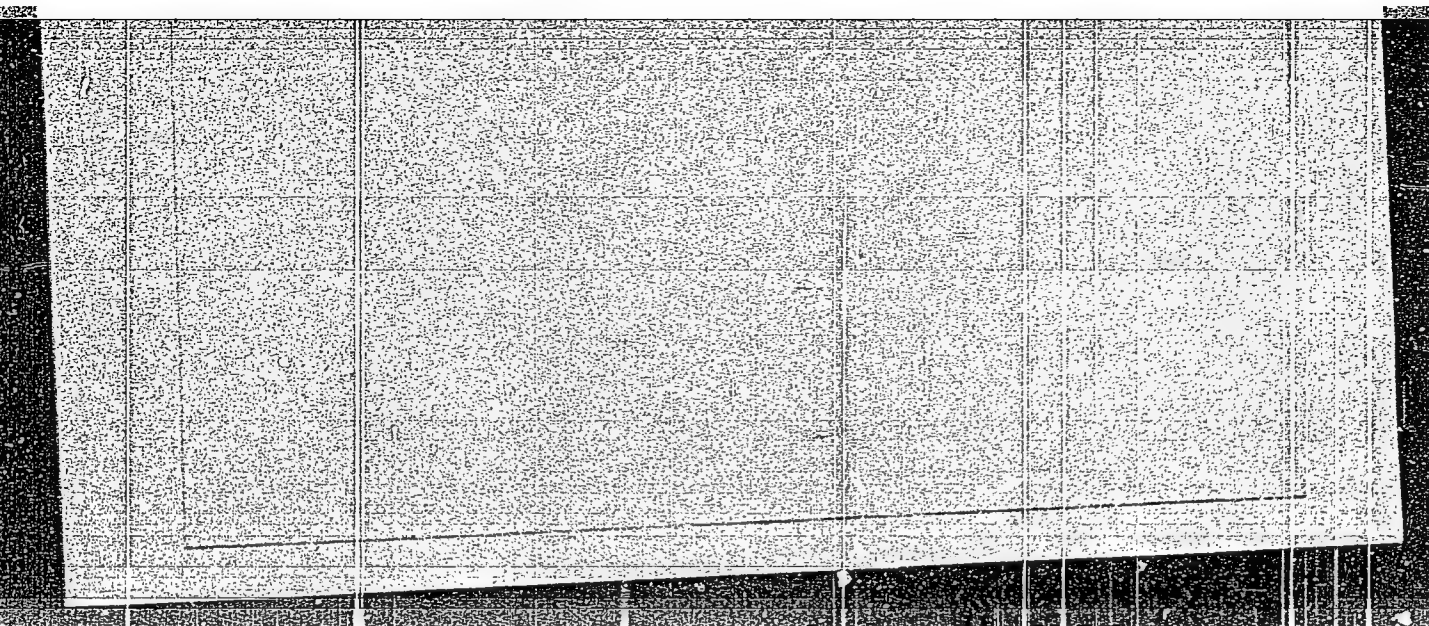
Chow
~~p-Nitroacetophenone~~ Olo Wichterle and Pavel Cechin.
 Czech. 84,638, Oct. 1, 1955. ~~2,4,6-Trinitrophenol~~ (I) is obtained
 in a two-step reaction involving conversion of p-nitroacetophenone
 (II) to its hydroperoxide by autooxidation followed by de-
 compn. in the catalytic presence of Cu salts. In a cylindri-
 cal reactor O was bubbled at a rate 10 l./hr. and 125°
 through a mixt. of 82.2 g. II and 0.4375 g. Bz₂O₂ contg.
 traces of a satd. aq. soln. of NaOH. After bubbling through
 30 l., 50 l., and 70 l. O 3 times 1 ml. satd. aq. soln. of Cu-
 (NO₃)₂ was added at each interval. After cooling, the mixt.
 was extd. by shaking with three 15-ml. portions of dil.
 H₂SO₄ (1:5) and the aq. layer sepd. The oily residue was
 washed, dried and distd. at 6 mm., yielding 05.54 g. non-
 reacted II, b. below 135°, 4.99 g. I, b. 145-82°, and 3.12 g.
 yellow oil, b. above 160°. The conversion was 67%, the
 yields were 30.15%.

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Wichterle, O.

Chem
Depolymerization of polycaprolactam by alkali carbonates. O. Wichterle, J. Sebenda, and J. Kariček (Faserforsch. u. Textiltech., 1955, 6, 563-568). To test the feasibility of recovering monomeric caprolactams from polyamide wastes by catalytic depolymerization of the polyamides, an experimental investigation is made of the depolymerization of pure polycaprolactam on heating with varying amounts of Na_2CO_3 (I), NaOH (II), and H_3PO_4 (III) as catalyst. In these tests a mixture of the polylactam and the reagent is heated at constant temp. (300° and 270°) under N_2 with the monomer distilling over as fast as it is formed. The best results are obtained with Na_2CO_3 , there being a high yield (~88-88.9%) of monomer with but little or no decomposition to by-products. With NaOH the velocity of depolymerization is much higher (4 times as high) but the yield of monomer is somewhat lower and there is considerable decomposition to unwanted by-products and the quality of the monomer is not so good. With H_3PO_4 the yield of monomer is much lower (~64-67%) and strong decomposition of the polyamide occurs. In all cases there is sublimation of a little dimer. The characters of the residues remaining after the monomer has been distilled off is described, and optimum amount of Na_2CO_3 for the depolymerization is the same as the optimum for the catalytic polymerization of monomeric caprolactam to polyamide. In applying the depolymerization with Na_2CO_3 to mixed lactam polymers (caprolactam/hexamethylenedipamide copolymers) it is found that the lactam is selectively and exclusively depolymerized to monomer, the reaction occurring rapidly and quantitatively. Thus a simple process is provided for the quantitative estimation of caprolactam in lactam mixed polymers.
H. L. WHITEHEAD.

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M. A. YOUTZ
2 copies

PM 1954

O. WICKTERLE

✓ 13932* A Pneumatic Valve for Measuring Liquid Doses. Ein pneumatisches Ventil für die Dosierung von Flüssigkeiten. (German.) J. Pinkava and O. Wickterle. Collection of Czechoslovak Chemical Communications, no. 3, June 1955, p. 697-707.

① Versatile dosing valve in which the flow, controlled by gas pressure, can be adjusted to regulate the flow of any liquid or liquid mixture of constant composition according to pressure conditions or density and viscosity of the liquid. Diagrams, graph. 4 ref.

ESP

WICHTERLE, O.

Czechoslovakia/Organic Chemistry - Synthetic Organic Chemistry, E-2

Abst Journal: Referat Zhur - Khimiya, No 19, 1956, 61471

Author: ~~Wichterle, O.~~, Cerny, J.

Institution: None

Title: Chloromethylation of Styrene

Original
Periodical: Sb. chekhosl. khim. rabot, 1955, 20, No 6, 1288-1291; German;
Russian

Abstract: See Referat Zhur - Khimiya, 1956, 32424

Card 1/1

Wichterle, Ota

5

Chloromethylation of styrene. Ota Wichterle and Jitka Cerny (Vysoka škola chem. technol., Prague). *Chem. Zvesti* 49, 1038-40 (1965). $\text{PhCH}=\text{CH}_2$ (I), CH_3O , and HCl form none of the expected *o*- or *p*- $\text{ClCH}_2\text{CH}(\text{CH}_3)\text{CH}_2\text{CH}_2$, but give $\text{PhCH}=\text{CHCH}_2\text{Cl}$ (II) in good yields. Heating a stirred mixt. of 78.1 g. I, 75 g. 20% aq. CH_3O , 375 ml. 37% HCl , and 1 g. S (as inhibitor of polymerization of I) 6 hrs. at 80° , sepg. the oily layer, dilg. the aq. layer with an equal vol. H_2O , extg. with two 100-ml. portions of Et_2O , washing the oily layer combined with the ext. with 50 ml. H_2O , twice with 50 ml. 10% Na_2CO_3 , drying with CaCl_2 , and distg. in vacuo after the addn. of 1 g. S yielded 38.1 g. II; b_p 114-16°, d₄ 1.0958, n_D 1.6783. Refluxing 28.16 g. II 2 hrs. with 35.12 g. hexamethylenetetramine in 250 ml. EtOH , filtering the crystals, and decompg. them by refluxing 2 hrs. with 500 ml. H_2O gave after ether extn. 0.4 g. $\text{PhCH}=\text{CHCHO}$, b_p 128-30°, n_D 1.6191; phenylhydrazone, m. 108-9°. The following compds. were prepd. from II: $\text{PhCH}=\text{CHCH}_2\text{OH}$, b_p 142-5°, d₄ 1.0442, n_D 1.5810; $\text{PhCH}=\text{CHCH}_2\text{OAc}$, b_p 101-2.5°, n_D 1.5451; $\text{PhCH}=\text{CHCH}_2\text{OAc}$, b_p 145-6°, d₄ 1.0560, n_D 1.5425; $\text{PhCH}=\text{CHCH}_2\text{SH}$, b_p 124-5°, n_D 1.0040 (isothiocyanate salt, m. 170-1° (from EtOH)). M. Hudlicky

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①

Wichterle, Oto.

✓ Methyl ester of 1-naphthylacetic acid. Oto Wichterle and Jiri Cerný. Czech. 85,459, Jan. 1, 1960. A new synthesis is described proceeding via $C_{10}H_8$ (I), $1-C_{10}H_7CH_2Cl$ (II), $1-C_{10}H_7CH_2CN$ (III), and $1-C_{10}H_7CH_2C(=NH)OEt.HCl$ (IV) to the Me $1-C_{10}H_7CH_2CO_2H$ (V) known as a phytohormone. The over-all yield of V, based on I, is 70%. Crude II (77.26 g.) (contg. 61.98 g. pure II, obtained in 92.6% yield by heating 64 g. I, 60 g. 24.91% HCHO, and 600 ml. 37.03% HCl 6 hrs. to 70° with vigorous stirring) treated with 28.56 g. KCN and 138.9 g. MeOH, refluxed 10 hrs. with vigorous stirring, HCl gas passed into the mixt. (contg. 44.18 g. III in MeOH) 3 hrs. at a rate 13.2 l./hr. with stirring and cooling to 20°, the resulting IV hydrolyzed by the addn. of 390 ml. water, and the product extd. with C_6H_6 and distd. yielded 47.52 g. V, b, 165-7°, n_D^{20} 1.5985. L. J. Urbánek

WICHTERLE, O. (Prof., Dr.)

Czechoslovakia

Neure Entwicklung auf dem Gebiet der Theorie und Praxis der Hochpolymeren

(Hauptjahrestagung 1956 der Chemischen Gesellschaft in der Deutschen Demokratischen Republik).

AUS DEM TAGUNGSPROGRAMM - Nachmittags: Gruppe C:

Prof. Dr. O. WICHTERLE, Prag, "Über die anionische Kaprolaktampolymerisation."

SOURCE: Plaste und Kautschuk, October, 1956, Unclassified.

- WICHTERLE, O.

CZECHOSLOVAKIA/Chemistry of High-Molecular Substances

F.

Abs Jour : Referat Zhur - Khimiya, No 2, 1957, 4636

Author : Wichterle, O., Sebenda, J.

Inst :
Title : Polymerization of Epsilon-Caprolactam by the Action of Alkali Carbonates. I. Rapid Polymerization of Epsilon-Caprolactam by the Action of Sodium Carbonate.

Orig Pub : Sb. chekhosl. khim. rabot, 1956, 21, No 2, 312-317

Abstract : See RZhKhim, 1956, 54678.

Card 1/1

- 6 -

WICHTERLE, O.; EXNER, O.

Reaction of some unsaturated sulfonic acids with halogens. p. 922. (Chemické Listy, Praha. Vol. 50, no. 6, June 1956.)

SO: Monthly List of East European Accession (EEAL) LC, Vol. 6, no. 7, July 1957. Uncl.

OTO WICHTELE & PAREK CEFELIN

the oily layer with H_2O , and distg. it after drying gave 0.5 g. recovered I, 5 g. IV, b. 145-7°, m. 79°, and 3.1 g. V. Stirring 4 g. II, 80 ml. H_2O , and 0.7 g. $Cu(NO_3)_2 \cdot 3H_2O$ 2 hrs. at 97-8°, extg. the mixt. with Et_2O , evapg. the ether, and cooling the residual oil (3.68 g.) to -10 to -20° gave, after washing with $EtOH$, 1.73 g. IV, and after distn. of the mother liquor, V, b. 152-60°. Treating a dried mixt. of 22 ml. of a soln. contg. 6.01 g. II in I, and 10 ml. H_2O with a soln. of 8.50 g. $K_2Cr_2O_7$ in 45 ml. H_2O at 20°.

~~OTTO~~ WICHTERLE, Oto

CZECHOSLOVAKIA/Organic Chemistry - Synthetic Organic
Chemistry.

G-2

Abs Jour : Ref Zhur - Khimiya, No 8, 1958, 25211

Author : Wichterle Oto, Gregor Vojtech

Inst :

Title : Addition of Dienes to Geminal Cyan-Nitroso-Compounds.

Orig Pub : Chem. listy, 1957, No 4, 605-611

Abstract : 2,3-Dimethyl-butadiene-(1,3) (I) does not react with nitro-
soisobutane (II). An equimolecular mixture of I and II or
a solution of I and II in C_6H_6 or ether remain unchanged
after 3 months or after one year (in a sealed vial). From
200 g 84% HCN (III) and 100 g acetone-oxime are synthesi-
zed 35 g alpha-hydroxylamino-isobutyronitrile, MP 100°
(from petroleum ether-ether) which in aqueous solution.

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Abs Jour : Ref Zhur - Khimiya, No 8, 1958, 25211

BP 90-91°/40 mm, and a certain amount of tris-(2-cyan-2-propyl)-hydroxylamine, MP 79-80°. Moist IV and butadiene-(1,3) (VII), in C_6H_6 (20°, 24 hours), form 2-(2-cyan-2-propyl)-3,6-dihydro-1,2-oxazine (VIII), yield 71%, BP 69-69.5°/1 mm, 76-78°/3 mm, n_{20}^D 1.4689, d_4^{20} 1.028. VIII is also formed on allowing 3,6-dihydro-1,2-oxazine (IX) and acetone-cyanohydrin to stand for 5 days, yield 87%. Analogously to VIII is synthesized from dry VI and I the 2-(1-cyan-cyclohexyl)3,6-dihydro-4,5-dimethyl-1,2-oxazine, yield 60%, MP 111.5-112° (from CH_3OH). If no solvent is used a vigorous reaction takes place and the substance is decomposed with formation of tris-(1-cyan-cyclohexyl)-hydroxylamine (X) which is difficult to isolate. Analogously to VIII is obtained, from moist VI and VII (12 hours), 2-(1-cyan-cyclohexyl)-3,6-dihydro-1,2-oxazine (XI), yield 81%, BP 155-156°/12 mm, MP 53.5°.

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is obtained thereafter (5-15°, 6 days) 2-(1-cyan-

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Chemistry.

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cyclohexyl)-tetrahydro-1,2-oxazine, yield 52%, BP 117°/
3 mm, MP 43.5° (from CH₃OH or hexane), n_{D}^{20} 1.4869,
which is also formed on hydrogenation of 10 g XI over Pt
in CH₃OH, yield 9.37 g. Analogously a hydrogenation of
5.55 g XIV produces 5.3 g 2-(1-cyan-cyclohexyl)-3(6?)-
methoxy-tetrahydro-1,2-oxazine, MP 64-65° (from CH₃OH).
On the basis of the results of the present and previous
researches (see RZhKhim, 1954, 46326) a rule is formula-
ted concerning the capacity of nitroso-compounds to under-
go diene-addition: in the aliphatic series the dienophi-
lic nature is exhibited only by those nitroso-compounds
from the nitroso-group of which electrons are drawn off by
the action of a proximal strongly polar linkage (C-Cl, C≡
C). An analogous phenomenon is observed in the case of
aromatic nitroso-compounds, wherein the free pair of

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Chemistry.

G-2

Abs Jour : Ref Zhur = Khimiya, no 8, 1950, 25011

electrons at N is drawn off as a result of conjugation
of nitroso-group and aromatic ring.

Card 6/6

WICHTERLE, O.; ZELINGER, J.

A method for preparing linear density gradients and their use in polymerization studies.

P. 265. (Chemicky Prumysl.) (Praha, Czechoslovakia) Vol. 7, No. 5, May 1957

SO: Monthly Index of East European Accession (EEAI) LC. Vol. 7, No. 5, May 1958

WICHTERLE, O. : CEFELIN, P.

"Preparation and decomposition of p -intro- α -cumylhydroperoxide.
In German."

p. 274. (COLLECTION OF CZECHOSLOVAK CHEMICAL COMMUNICATIONS. SBORNIK
CHECKHOSLOVATSKIKH KHMICHESKIKH RABOT. -- Praha, Czechoslovakia.)
Vol. 22, No. 1, Feb. 1957

SO: Monthly Index of East European Accession (EEAI) IC, Vol. 7, No. 5, May 1958

WICHTERLE, O.

Present state of development in the field of polymeriza-
tion of carboxylic acids. O. Wichterle (Tech. Hochschule
Chem., Prague). Collection Czech. Chem. Commun. 22,
Spec. Issue, 233-82, discussion 292-4 (1957) (in German).
A review with 15 references. C. E. Feast

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WICHTERLE, O.

CZECHOSLOVAKIA/Chemistry of High-Molecular Substances.

I

Abs Jour: Ref. Zhur-Khimiya, No 11, 1958, 38492.

Author : Wichterle O., Sebenda J.

Inst : Not given.

Title : Polymerization of ϵ -Caprolactam by the Action of Alkali Carbonates. II. The Kinetics and Mechanism of Alkali Polymerization of ϵ -Caprolactam Sb chekhosl khim rabot, 1957, 22, No 5, 1353-1367.

Abstract: See RZhKhim, 1956, 65167.

Card : 1/1

mole for I and 27.2 kcal/mole for II. Logarithm of the quantity preceding the exponent is 9.56-10.21 for I and

CZECHOSLOVAKIA/Physical Chemistry - Kinetics, Combustion.
Explosions, Topochemistry. Catalysis.

B-9

Abs Jour : Ref Zhur - Khimiya, No 8, 1958, 24214

11.08-11.40 for II, depending on the concentration of
 H_2SO_4 (0.1-0.4 M). Velocity constants are a linear func-
tion of H_2SO_4 concentration.

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WICHTERLE, OTTO

CZECHOSLOVAKIA/Laboratory Equipment, Apparatus, Their
Theory, Construction and Application.

F.

Abs Jour : Ref Zhur - Khimiya, No 14, 1958, 46532

Author : Otto Wichterle, Otokar Mikeš

Inst : -

Title : Simple Instrument for Countercurrent Separation.

Orig Pub : Chem. listy, 1957, 51, No 8, 1569-1574

Abstract : The construction of a laboratory instrument for counter-current separation of liquids is described. The instrument consists of a series of cells - glass tubes of a special shape - fixed on a common pivot stand. Several schemes of cell connection and an example of the instrument application are presented.

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CZECHOSLOVAKIA/Chemistry of High-Molecular Substances.

I

Abs J⁰ur : Ref Zhur - Khimiya, No 17, 1958, 59749
 Author : Wichterle Ota, Zelinka Jiri
 Inst :
 Title : Copolymerization of Different Vinylidenehalides.
 Orig Pub : Chem. listy, 1957, 51, No 11, 2146-2148.

Abstract : Systems of 1-chlor-1-bromethylene (I) - 1,1-dichlorethylene (II) and 1,1-dibromethylene (III)-II were investigated. The composition of the copolymers was found by analytical determination of halogens with an accuracy of $\pm 0.1\%$. The following values were obtained for the constants of copolymerization: for I-II, $r_1 = 2.38 \pm 0.06$, $r_2 = 0.83 \pm 0.08$; for III-II, $r_1 = 1.90 \pm 0.11$, $r_2 = 1.04 \pm 0.10$. Monomers were preserved for the prevention of spontaneous polymerization in 50% alcohol solutions.

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